



NTNU – Trondheim
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Science and Technology

NEW AND ENERGY EFFICIENT DRYING FOR PROTEIN MIXTURES

Treatments and drying conditions effects on
product quality and kinetics

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MASTER THESIS

for

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NEW AND ENERGY EFFICIENT DRYING FOR PROTEIN MIXTURES
- Treatments and drying conditions effects on product quality and kinetics

Background and objectives

Drying is an essential process applied in most industry to remove moisture and extend the shelf life of final dried products without refrigeration. However, drying is an energy intensive process taking up to twenty of the energy used in the industrial sector. There is large energy losses in the currently used conventional dryers resulting in high operation costs, harm to environment and instability to climate. With the certainty of persistent rise in fuel and energy costs the challenges are to develop technologies that are energy efficient and minimize impact to environment and climate.

When applied to feed and mixtures of protein, carbohydrate and lipid (PCL) the main drawbacks of the conventional dryers are low quality and undesired properties of the final dried product.

A sustainable and advanced heat pump drying technology has been tested and developed at NTNU. This drying technology will be applied to protein mixture an alternative to solve problems of the conventional dryers. The heat pump drying technology is energy efficient and environmentally-friendly and has the added benefits of competitive costs and high quality final dried products.

The objectives are:

- to conduct experiments in drying of two types of protein mixtures (PM)
- to identify a range of temperatures capable of producing dried protein mixtures
- to study how the quality and properties of the dried product are affected by: mixture composition , boiling treatment, mixture phase, particle size and temperature or relative humidity.

Experimental design: The following experiments will be done on a lab scale heat pump dryer using standard and standard plus carbohydrate, frozen and unfrozen mixture, boiled in spherical shape with size ranging from 1 to 12 mm, and unboiled mixture, cube sizes of 10 and 20 millimeter and air temperatures of -1 and 20°C.

The experimental design with mixture composition, treatment, geometry and drying temperature

Test	*RC	*BO	*MP	Cube length, mm	t, °C
1	-	+	-	1 to 12	20
2	-	+	+	1 to 12	20
3	-	-	-	10	-1
4	-	-	-	20	-1
5	+	-	+	20	20
6	+	-	+	20	30
7	+	-	+	10	20
8	+	-	+	10	30

* RC: - for standard mixture and + carbohydrate addition, BO: - unboiled and + boiled , MP: - frozen and + unfrozen.

The main experimental tasks are:

1. to review the related literature
2. to thermal treat PCF samples 1 and 2
3. to carbohydrate to samples 5 to 8
4. to perform heat pump drying tests of samples 1 to 8
5. to collect analyze the data on quality, properties and kinetics
6. to write and submit the thesis according to the deadline

The Master thesis work comprises 30 ECTS credits.

A progress plan (*planned activities and scheduled progress*) shall be submitted to the responsible subject teacher/supervisors for comments within 14 days after the candidate has received the project description.

The work shall be edited as a scientific report, including a table of contents, a summary in Norwegian, conclusion, an index of literature etc. When writing the report, the candidate must emphasize a clearly arranged and well-written text. To facilitate the reading of the report, it is important that references for corresponding text, tables and figures are clearly stated both places.

By the evaluation of the work the following will be greatly emphasized: The results should be thoroughly treated, presented in clearly arranged tables and/or graphics and discussed in detail.

The candidate is responsible for keeping contact with the subject teacher and teaching supervisors.

Risk assessment of the candidate's work shall be carried out according to the department's procedures. The risk assessment must be documented and included as part of the final report. Events related to the candidate's work adversely affecting the health, safety or security, must be documented and included as part of the final report.

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The report shall be submitted to the department in 3 complete, bound copies.

An executive summary of the thesis including title, student's name, supervisor's name, year, department name, and NTNU's logo and name, shall be submitted to the department as a separate pdf file. Based on an agreement with the supervisor, the final report and other material and documents may be given to the supervisor in digital format.

Submission deadline: June 16, 2012.

Department for Energy and Process Engineering, 16 June 2012.



Olav Bolland
Department Head



Odilio Alves-Filho
Supervisor

Abstract

Drying is an industrial process widely used to extend the shelf life of products. Heat pump drying technology gives final dried products with better quality without the main concerns of the conventional dryers such as environmental challenges and large energy losses that lead to higher operational costs. Eight tests involving two different types of protein mixtures were carried out at the Norwegian University of Science and Technology in Trondheim, Norway. The study covered the influence of parameters like mixture composition, boiling treatment, mixture phase, particle size and temperature or relative humidity on the quality and final properties of the dried products. Obtaining the drying kinetics it was done to evaluate the best conditions for a better water removal from the product. The content of carbohydrate improved the drying rates thus allow to make a lower cost product.

Acknowledgements

The carrying out of this thesis would not have been possible without the support of every people who have been with me during all my life.

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I would like to specially thank my parents and my sisters for being with me every time I needed it.

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”Anyone who has never made a mistake has never tried anything new.”

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Nomenclature

Abbreviations

CFC	Chlorofluorocarbons
CH	Carbohydrate
COP	Coefficient of performance, dimensionless
dh/dx	Thermal efficiency of the dryer (kJ/kg)
HPD	Heat pump drying
SMER	Specific moisture extraction ratio (kg/kWh)
STD	Standard

Symbols

ε	Porosity
ρ_b	Bulk density (kg/m ³)
ρ_p	Particle density (kg/m ³)
Q_0	Refrigerating capacity (kW)
S_b	Shrinkage
V	Final volume (m ³)
V_0	Initial volume (m ³)
W	Energy consumption or compressor mechanical work (kW)
X_{db}	Moisture content, dry basis
X_{wb}	Moisture content, wet basis
ϕ	Relative humidity

Chapter 1

1. Introduction

Since former times drying has been used as a method to preserve goods and specially biomaterials. Conventional dryers need a large amount of energy having a negative impact in the environment and increase in pollution that is one of the main concerns of these days. Also because of the high temperatures used the product may experience changes and detrimental effect on quality and properties. Thus, final product value in the market decreases. For these reasons extensive investigation has been made and some technologies have been developed in order to find out the better processes and conditions.

Freeze drying has been studied as an alternative to the traditional dryers. With this technology the product damage caused by the temperatures applied is reduced considerably. It can be carried out under very low temperature and vacuum requiring high investments in machinery. Because of this, it is only profitable for high value goods such as pharmaceuticals.

The atmospheric freeze drying process offers an economical solution to dehydrate products while keeping its quality. The limitation of this method is that it requires a long drying time. Combining atmospheric freeze drying with a heat pump system increases the thermal efficiency as well as it enhances the physical properties of the dried product. Heat pumps allow working with a wide range of air temperatures and humidities giving more flexibility to adapt this method to different kinds of materials and products. To decrease even more the drying time accelerating processes can be applied. Ultrasonics, vibrated fluid beds, microwaves and infrared radiation are some of them.

Chapter 2

2. Literature review

Heat pump is used for drying biological materials due to its many advantages. It is an efficient energy and environmentally friendly technology that allows obtaining good quality products by operating with adjusted and controlled conditions.

The value of the final dried product is highly dependent on quality and properties such as size, shape, color, density and drying kinetics. The conventional drying process takes large amounts of energy and it influences negatively in the environment and climate changes.

ENERGY

Alves-Filho and Strømmen [18] mentioned that Heat Pump Drying (HPD) technology requires 20% to 40% of energy when compared to the traditional dryers at the same temperature. Perera and Rahman [19] established the differences in SMER (0.1-1.3, 0.7-1.2 and 1.0-4.0kg/kWh) and efficiency (35-40%, less than 70%, 95%) between hot air drying, vacuum drying and HPD respectively. The energy efficiency of the heat pump system also increases significantly with the number of stages [12]. For a two-stage compressor driven heat pump dehumidifier with subcooling a COP of 8.0 can be reached (Perry, 1981). HPD with the combination of technologies such as intensification by ultrasound, microwave, infrared radiation and vibrated fluid bed [6, 7, and 8] has higher drying rates and lower energy consumption.

QUALITY PARAMETERS

During drying important physical, chemical or nutritional and microbiological degradation may take place in the product. These yield poor quality properties making a final product that is less valuable. The most important quality and properties indicators are the moisture content, density, rehydration and color. Many studies about HPD have

shown that under determined conditions food properties improved compared to traditional drying technologies.

- **Moisture content**

Drying kinetics are greatly influenced by parameters such as air conditions and sample characteristics, geometry and size. Green beans, potatoes and peas were shaped as parallelepiped, cylindrical and sphere with different sizes. Such products were tested by Wijitha Senadeera et al. under three different temperatures (30, 40 y 50°C) using a fluidized bed dryer with a heat pump system. It was found that as the size increased, the drying rate decreased for every temperature in the tests. The drying temperature was the most important factor affecting the drying rates [21]. It was also corroborated by M.K. Krokidaa et al. through experiments performed with various plant materials such as potato, carrot, pepper, garlic, mushroom, onion, leek, pea, corn, celery, pumpkin and tomato. The effect of air velocity and air humidity was considered lower than that of air temperature for tested products [20].

- **Bulk density**

Odilio Alves-Filho and Ingvald Strømmen [4, 5] conducted tests involving potatoes, fruits, cod fish, fish feed and shrimps. For 5 mm cubic particles of cod-fish dried from -10°C to 30°C in a HPD, the bulk density decreased as the temperatures were lowered and the time period increased. The bulk density is also influenced by the dry matrix of the product as it can be inferred comparing extruded beate, beate cubes, beate potato powders and the commercial products. Myosin protein particles were tried by Odilio Alves-Filho et al. The measured values indicated that using atmospheric freeze drying yields lower bulk densities and highly porous dried materials. It was also found that an increment in the residence time at lower temperature causes a drop in bulk density.

- **Color**

The work done by Julia Heizinger [8] with green peas shows the different quality values achieved after 6 hours of drying with HP at three different temperatures (-6°C, -3°C and 0°C). Similar values to the initial ones were obtained for lower temperatures. Red peppers were also tested at temperatures of -10, -5, -3 and 20°C by Odilio Alves-Filho et al. [23]. The best color parameters were obtained for the run done at -10°C with

values closer to the initial ones. Despite of this, the trial carried out at 20°C gave also favorable results demonstrating that a wide range of temperatures can be used in HPD without damaging the product quality.

- **Rehydration**

The rehydration capability is highly related to the density and porosity of the dried product. It was seen that for four different classes of potatoes, subjected to variable drying conditions the rehydration index was inversely proportional to the bulk density [5]. It was highest for the extruded beate, followed by the non-extruded beate, pimperl and mandel potato varieties. In experiments in which red peppers were tested under different temperatures, it was found that better rehydration rates were obtained for samples dried at lower temperatures. By immersing the product during 60 s in a 90°C water bath higher rates were achieved than using water at 20°C [23].

This work covers single atmospheric freeze drying of mixtures of proteins and carbohydrates with a heat pump system. The main objective is to evaluate the quality changes and the drying rates achieved by using this technology. The kinetics, as well as the color, density and rehydration capability will be investigated in this work. Different raw materials, temperatures and shapes will be used to perform eight tests carried out in the laboratory heat pump dryer designed and built at the Norwegian University of Science and Technology. The materials used are protein and carbohydrate mixtures. The drying test batches were prepared with protein in its natural form, boiled or mixed with carbohydrate. The boiled material was split in samples that were frozen and unfrozen and the tests were made with an air temperature of 20°C. The standard material was cut into cubic particles of 10 and 20 mm to produce two batches that were subjected to an air temperature of -1°C. Finally, the runs involving mixture of protein and carbohydrate were performed at air temperatures of 20°C and 30°C using cubic samples of 10 and 20 mm side.

Chapter 3

3. Theory

3.1 Dehydration

Dehydration or drying is a method based on the removal of water by evaporation or sublimation under controlled conditions. The processes involve heat and mass transfer. Heat transfer may occur by conduction, convection or radiation or combination of these mechanisms, which are based on the temperature differences between the material and the transport fluid or heating source. Mass transfer is caused by other driving forces such as vapor pressure gradients.

The main aims of drying technologies are:

- Improve the preservation of foods
- Extend the shelf-life of products
- Reduce weight and volume to lessen costs in transport
- Reduce the environmental impact
- Provide new products with new characteristics

Despite this, the principal drawback of using conventional drying is the changes in quality parameters such as flavor, color, aroma, taste, texture or nutritive values. To avoid this, lower temperatures and higher drying times should be applied. One important aspect that occurs during drying is shrinkage. If the amount of water removed is lower than corresponding pores or shrinkage, some forces can appear causing deformation and leading to a structural collapse. Case hardening, surface cracking and loss of rehydration are among the main effects of this phenomenon [3].

To design a better drying system it is necessary to obtain data on drying kinetics. The curve shown in Figure 1 represents the drying rate versus the moisture content of the product. The drying rate curve can be divided in three different regions [1]:

- I. Rising rate, which is a short period where the drying rate increases as the moisture content lowers. During this phase the product progressively loses moisture.
- II. Constant rate in which the drying rate remains almost constant until the moisture content reaches a determined value called critical moisture content. In this region the rate of evaporation is higher than the water moving to the surface.
- III. Falling rate, where the drying rate decreases rapidly as drying time approaches a large value.

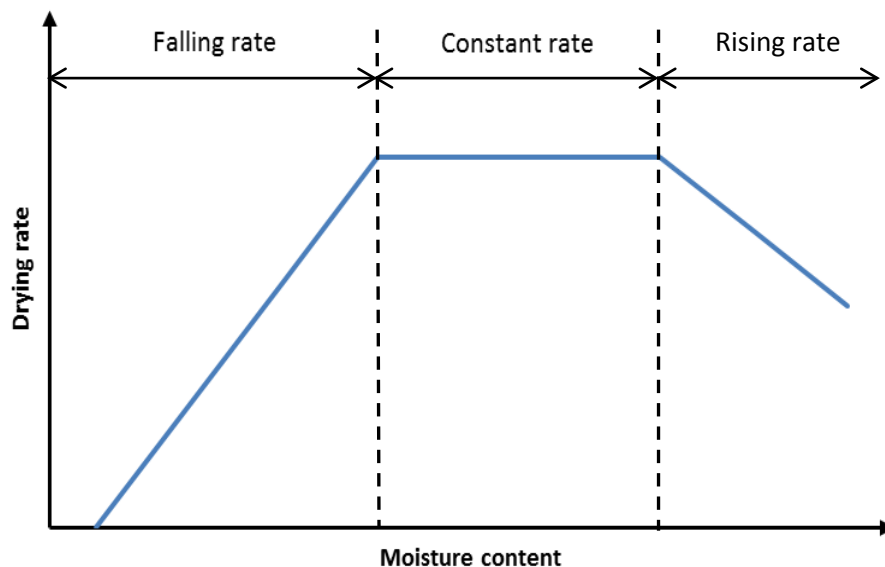


Figure 1. Drying rate curve

The main parameters that affect the drying rate and capacity can be classified as internal and external factors as follows [1]:

- Internal factors
 - Shape
 - Size
 - Porosity
 - Moisture.

- External factors
 - Air temperature that has major effect during the constant phase. Larger temperatures involve lower drying times.
 - Air velocity, which an increasing value leads to higher rates of heat and mass transfer enhancing the drying rates. It has small or no effect in the falling rate period.
 - Air humidity, that when increased causes a reduction in the drying rate with major influence in the constant rate period.

3.1.1 Dryers in the food processing industry

According to Zeki Berk [1], the dryers used in the food industry can be classified as follows:

- **Cabinet dryers**

This inexpensive technology is used when moderate amounts of solid foods are dried in batch mode. The material is placed on trays inserted into shelves distributed inside a drying chamber. The hot air flow passes through trays to remove moisture from the product. Due to the arrangement of the material, the moisture content distribution is variable and dependent on the position of the tray. To avoid this, solutions are proposed based on test measurements. Cabinet dryers have been used to dry some types of fruits, vegetables and herbs with a temperature range from 60 to 80°C during 2 to 10 hours. Recirculation of the air may lead to lower energy costs.

- **Tunnel dryers**

Tunnel dryers are used for continuous drying of foods in solid state. Many trays carried by trolleys move through long tunnels in which the product is gradually dehydrated. The air flow and tray relative movement can be co-current, countercurrent or mixed. The co-current flow provides higher drying rates at the entrance of the tunnel while the countercurrent allows drying the material at the lower moisture content. The mixed type combines both advantages. In these dryers the external conditions can be variable.

- **Belt**

Belt dryers are designed to dry solid and paste foods such as fruits, vegetables and tomato in a continuous mode. The material is moved on conveyor belts. Single-stage or multistage are available with cross-flow, through-flow or a mixture flow. For this kind of dryers the product load and discharge operations are easier than in the previous cases. Moreover, the moisture content is more homogeneous and higher loading can be achieved.

- **Belt-trough dryers**

Belt-trough dryers are widely utilized to dry vegetables in a continuous operation. The food is placed on a wide mesh belt that is supported by two rollers. The material is mixed during the movement and as hot air flow passes through the dryer. The water removal is relatively rapid and usually occurs in less than one hour.

- **Rotary dryers**

Waste materials and animal feed are usually dried using this kind of dryers. An inclined metal cylinder rotates over supporting roller. The material inside may climb through the drying chamber and after reaching a determined angle it may drop to the bottom again or be discharged. The flow may be co-current or countercurrent and the air flow is blown through the cylinder and material to be dried. If the temperature of the air is too high rotary dryers can function as roasters.

- **Bin dryers**

Bin dryers give the solution to achieve lower final moisture contents that cannot be reached with tray, belt and belt-trough dryers. The air flows along a container that is perforated in the bottom, inside of which the material is placed. The moisture distribution is uniform due to the internal transport and the large residence time.

- **Grain dryers**

Dryers for grain and seeds use air temperatures between 50 and 70°C to obtain moisture contents around 12 to 15% wet basis (wb). These dryers can function in batch or continuous modes. For batch dryers, the upper section is pre-drying to avoid the differences in moisture content within the product. In continuous dryers the air flows through the moving grains or seeds. To prevent heat damage cool air is blown across the material. The drying rates achieved are high.

- **Spray dryers**

Spray dryers are used in continuous mode to dry liquid, solution and paste foods to obtain powder products like powder milk, instant coffee and tea. The material that is going to be dried is atomized or sprayed into high temperature air flowing through the chamber. The liquid droplets and the high air temperatures applied produce dried particles of powders. Due to the high drying rates achieved, the thermal damage caused by this technology may be low. A spray dryer is mainly composed of:

- Air heater
- Atomizer and nozzles
- Pump for feeding the liquid to the atomizer
- Drying chamber
- Solid-gas separators
- Fans to provide the air flow through the system

- **Fluidized bed dryer**

In this type of dryers, the hot air flow is used both for drying and fluidization. It may be utilized for fluidizable particles and relatively stick and non-sticky granules. The operation mode can be batch or continuous with high drying rates. Agglomeration and granulation can be done and stickiness can be avoided by application of vibrating fluid bed. Some of the products handled by this technology are vegetables, fruits, yeast particles and grains.

- **Pneumatic dryer**

This dryer has one stream of hot and dried air that flows through a loop and dries the particles moving within the loop. It is often used as a pre-treatment and followed by another drying process. It works in continuous mode and it is applied to dry flours, starch, gluten powder, etc.

- **Drum dryers**

Drum dryers can be classified as single and double-drum dryers. The latest type is composed by two drums that rotate in reverse directions keeping a flexible gap between them. The feeding process in single drum dryer involves applicator rolls, double-drum dryer with feed rolls, twin cylinder dryer with nip feed, single drum dryer with applicator roll and dip feed drum dryer. Some mechanisms are needed to ventilate and to remove the vapor from the vicinity of the drums dryer. It is used for liquid and paste products like mashed potatoes and dried soup.

- **Screw conveyor and mixer dryers**

Screw conveyors are used in continuous drying of solid and paste products as grains and wastes. The main heat mechanism is conduction. For batch drying of particles and powders mixers are built.

- **Sun drying, solar drying**

Sun drying is the dehydration of food using the heat radiation coming from the sun. It is mainly used for fruits, vegetables and fish. The term solar drying is related to the food dried with air previously heated using solar energy. The limitation of this technology is the season and daily discontinuity of solar radiation.

3.2 Atmospheric and vacuum freeze drying

Freeze drying can be done under different conditions of pressure and temperature.

First is the vacuum freeze drying that is a dehydration process where the water is removed from the product by sublimation at low temperature and vacuum. The products obtained by using this technology have high quality properties such as a rapid

rehydration, less shrinkage and better color. This is due to the lower temperatures applied that minimize the thermal damage. It can be done only below the triple point similarly and in accordance to the phase diagram of pure water shown in Figure 2 [1]. Vacuum freeze drying is only profitable for valuable goods. It is reported that it is about five times more expensive than conventional drying [2].

Second is the atmospheric freeze drying that is done at normal pressure but at different temperatures. This process applies to most types of products at lower cost than the previous process.

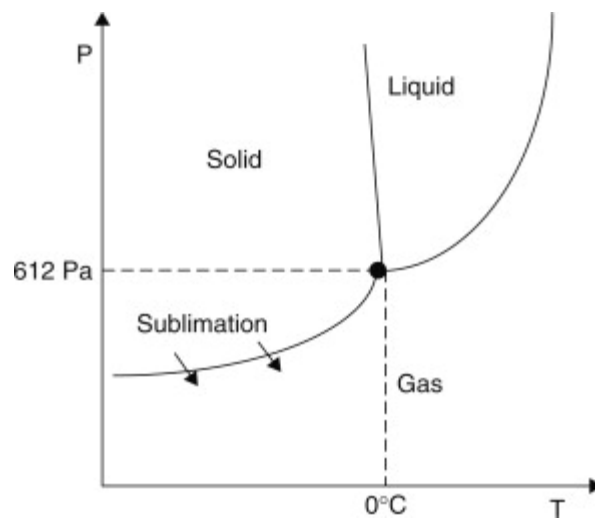


Figure 2. Phase diagram of pure water

An important drawback of freeze drying process, apart from the higher costs, is that it is slower than the traditional high temperature drying by evaporation. In freeze drying the water has to be first frozen before being transported as vapor during sublimation [2].

Before drying, the material is cut into pieces if it is solid or frozen and granulated if it is a liquid or semi-liquid. This is made to solidify the material and to increase the surface area to improve heat and mass transfer. The product can be either frozen inside or outside a chamber.

By controlling the drying conditions the drying time can be reduced as well as the quality is improved by avoiding structural collapse.

The components of a vacuum freeze dryer are the drying chamber, supporting elements, a source of heat, a refrigerated ice condenser and its refrigeration system, a vacuum pump to produce high vacuum and control and measurement instruments [1].

3.3 Heat Pump technology

Heat pump drying is an efficient and clean technology used to heat sensitive materials reaching good quality properties.

A simplified heat pump system is composed of an expansion valve, two heat exchangers (air cooler or evaporator and air heater or condenser) and a compressor. The sketch of the heat pump dryer and the diagrams of temperature-entropy and pressure-enthalpy are shown in Figures 3 to 5 [9].

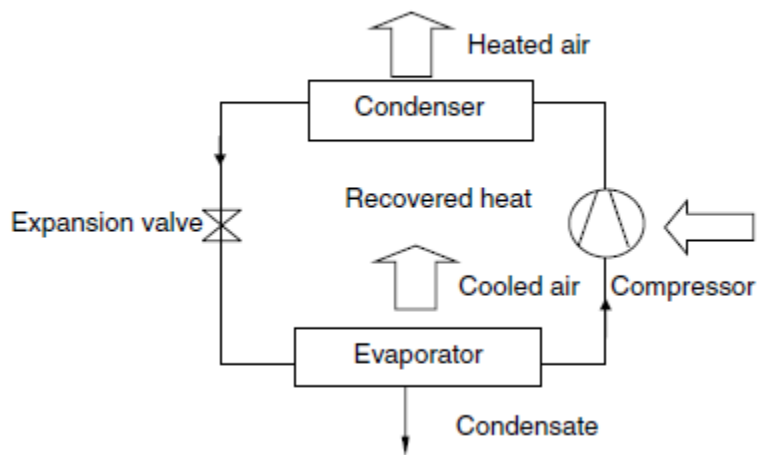


Figure 3. Heat pump system

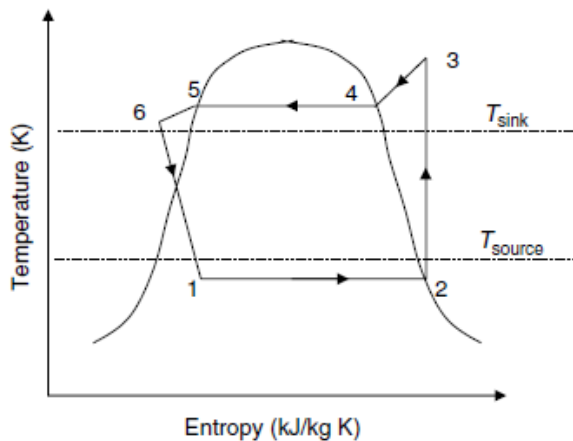


Figure 4. Temperature-entropy diagram

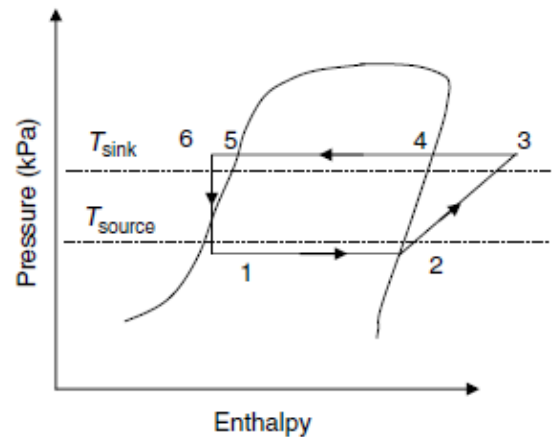


Figure 5. Pressure-enthalpy diagram

The drying air flows across the evaporator that contains a mixture of vapor and liquid refrigerant. The air flow releases heat and the refrigerant boils becoming saturated vapor in point 2. From point 2 to point 3 the compressor increases the refrigerant pressure with

the consequent elevation of the temperature. In point 3 the fluid is superheated vapor. By flowing through the condenser, the vapor is converted into saturated liquid at constant pressure (5). A subcooling state is reached in point 6. Finally, a throttling valve returns the liquid refrigerant at constant enthalpy to a vapor-liquid mixture at the initial point (1) starting the cycle again.

Some parameters to evaluate the efficiency of a heat pump system are the coefficient or performance (COP) and the specific moisture extraction ratio (SMER) expressed by the equations 1 and 2 [5]:

$$COP = \frac{\text{useful heat output}}{\text{power input}} = \frac{Q_0}{W} \quad (1)$$

$$SMER = \frac{\text{amount of water evaporated}}{\text{energy input to the dryer}} = COP \frac{dx}{dh} \text{ kg/kWh} \quad (2)$$

3.3.1 Advantages and limitation

The main advantages and limitations for a heat pump dryer are outlined next [9].

Advantages

1. SMER between 1.0 and 4.0 or higher can be reached due to the recovered heat from the moisture-laden air.
2. Improving of quality properties by applying lower temperatures. The same drying potential can be achieved by reducing the air relative humidity.
3. A wide range of temperature of -20°C to 100°C and relative humidity between 15% to 80% can be used.
4. Lower energy expenditure for low-value products and better drying conditions and control for the valuable ones.

Limitations

1. Some refrigerants such as chlorofluorocarbons (CFC) used in the cycle may not be environmentally friendly but there are natural fluids currently available.
2. Some maintenance is needed and also infrequent refrigerant charge.
3. It may involve higher costs than the traditional drying systems.

3.3.2 Design rules for heat pump dryers [5]

1. Continuous operation instead of batch mode that leads to a higher capacity and thermal efficiency.
2. Countercurrent operation (air and product) for tunnel heat pump dryers.
3. Inlet air temperature to the drying chamber as high as possible to increase the drying rates.
4. Avoid over sizing the refrigeration capacity that will reduce the SMER.
5. Assure the air flows through the product without possibility to by-pass
6. Select the best combination of evaporating and condensing temperatures for optimum COP and dh/dx .

Chapter 4

4. Material and experimental methods

4.1 Experimental laboratory single-stage atmospheric heat pump dryer

The equipment used to perform and carry out the tests was a laboratory heat pump dryer sketched in Figure 6.

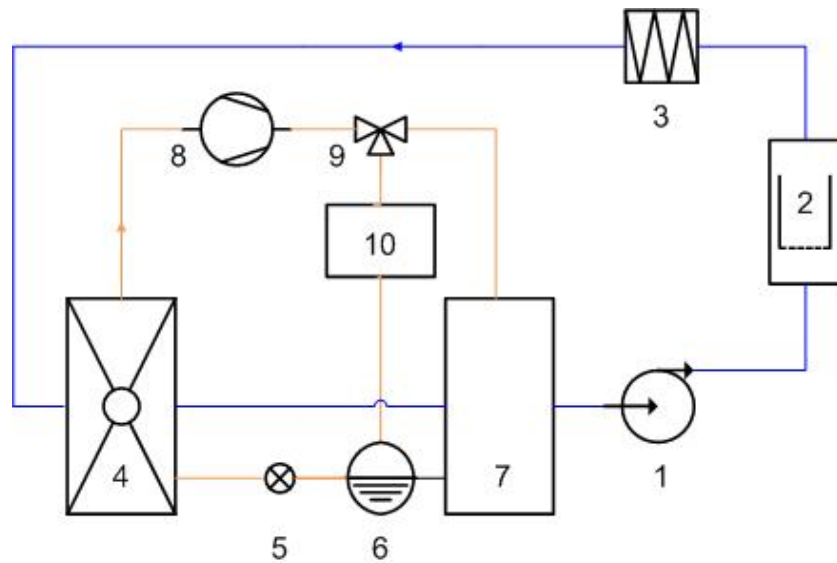


Figure 6. Experimental heat pump dryer. 1: Air blower, 2: Drying chamber, 3: Air filter, 4: Air cooler (evaporator), 5: Expansion valve, 6: Liquid receiver, 7: Air heater (condenser), 8: Compressor, 9: Three-way Valve, 10: Surplus heat exchanger

The air and heat pump loops are represented in blue and orange color lines respectively. The process begins when the air is blown to the drying chamber and flows through the product inside the drying chamber. Then, the air with increased humidity by taking moisture from the material is filtered and cooled in the evaporator. After being dehumidified the air flows and is heated in the condenser being ready to operate the loop cycle again.

Meanwhile, the refrigerant boils in the evaporator. Afterwards it is compressed, condensed and throttled by a three-way valve. The excess heat is removed by the surplus heat exchanger. After the expansion, the fluid mixture enters the evaporator where the cycle re-starts.

Figure 7 shows a picture of the drying cabinet in which the tests were performed. The dimensions of the drying cabinet are 80 cm width, 80 cm length and 1.5 m height. Figure 8 is a picture of the drying chamber that had a cylindrical shape with an internal diameter of 190 mm. It was made of Plexiglas and removable. A perforated plate was fixed on its bottom and a wire net was placed in the top of it to avoid possible mass losses due to the fluidization.



Figure 7. Drying cabinet



Figure 8. Drying chamber

4.2 Preparation of materials

The raw materials used to prepare the samples to perform the different tests were protein and carbohydrate, which derived from corn starch with 2% total carbohydrate supplied by Asia Food Import AS, Oslo. The total amount of protein processed was 10 kg while the quantity of corn starch was 260 g from which 5.2 g were carbohydrate.

4.2.1 Boiled samples

Before drying, 3000 g of protein were kneaded inside a metallic bowl and the mixture became homogenized. A sample of 2940 g of the initial product was placed into a deep pan and boiled during 15 minutes. After this heat treatment,

the final mass ready to run for the trials was 1940 g. The boiled material was deposited in a plastic vessel and dispersed with a long knife. To carry out the two first experiments, 875.9 g were stored in a freezer kept at -25°C and nearly the same amount was kept unfrozen in a fridge at higher temperature. Figures 9 and 10 show the raw material and the boiled material inside the deep pan.



Figure 9. Preparation of boiled material



Figure 10. Boiled material

4.2.2 Standard samples (STD)

The initial weight of protein in this case was 3500 g but the total mass of the batches used to perform these experiments was 3006.6g. After having a uniform mass, the samples were spread on a plastic board forming a layer of 14 mm thickness approximately. The whole slab was covered with a plastic film and carried to the freezer room. Once the product was frozen, it was taken out of the freezer and the film was split in two equal batches or sections. Using a caliber and a stainless knife, each portion was cut into 10 mm and 20 mm cube particles respectively. These samples were stored in the freezer.

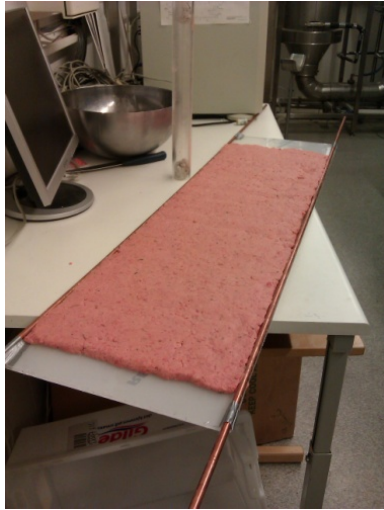


Figure 11. Preparation of STD material



Figure 12. STD material cubicles

4.2.3 Carbohydrate samples (CH)

The whole corn starch was mixed with 3500 g of protein with the aid of a mixer to produce the carbohydrate samples. Later, preparation of samples followed the same procedure. The material was kneaded by hand and spread on a plastic board. After being frozen, it was cut into pieces of 10 mm and 20 mm side and kept in the same freezer as the other samples.



Figure 13. Preparation of carbohydrate material

4.3 Drying methods

4.3.1 Heat Pump Drying

For every test except for the boiled samples, initial samples were taken to measure important parameters such as densities, color and moisture content.

Before each experiment, the conditions were set and the heat pump drying system was turned on. It took nearly one hour to reach stable set points. The drying chamber was weighed and loaded with the batch of each material. The sample mass was measured to determine the initial bulk density. Once the system was ready, the chamber was placed into the drying cabinet and the air velocity was regulated until proper fluidization was achieved. It depended on the bed of material and the type of drying material, which were STD, carbohydrate and boiled samples. The fluidization velocity was lower for the boiled samples. Every thirty minutes, during six hours and a half of drying, the air flow was reduced to take out the chamber and measure the mass lost using a digital scale. These losses were taken as the water removed considering a constant dried mass in the batch. For every run, except for the boiled samples, it was necessary to decrease the air velocity to avoid bubbling fluidization. The operation conditions including the air humidity, air and refrigerant temperatures in the inlet and outlet were monitored and recorded by a computer using a special program. Table 1 gives the main conditions for each experiment.

Run	Material	State	Shape [mm]	Initial mass [g]	T [°C]		φ		Air velocity [m/s]
					inlet	outlet	inlet	outlet	
1	Boiled	Frozen	Particles	875.9	20	16.7	19	34.9	3.60
2	Boiled	Unfrozen	Particles	876.2	20	15.7	9.5	17.6	3.60
3	STD	Frozen	20	1503.3	-1	1.6	37.5	42.0	5.50/5.20
4	STD	Frozen	10	1503.3	-1	1.0	37.9	43.7	5.50/5.20

5	CH	Frozen	20	854.7	20	17.8	20.9	26.4	5.15/4.80
6	CH	Frozen	20	854.6	30	24.9	7.1	10.8	5.15/4.80
7	CH	Frozen	10	854.6	20	18.4	16.9	20.3	5.15/4.80
8	CH	Frozen	10	854.6	30	26.1	7.4	10.8	5.15/4.80

Table 1. Experimental conditions

4.3.2 Oven Method

Three samples from each initial test were put on special metallic plates and dried in a conventional oven during a time interval of 14 hours at 130°C. After this period the samples were taken out to measure the mass loss in order to calculate the moisture content.



Figure 14. Oven

4.4 Measurements

4.4.1 Moisture content

The moisture content is one of the properties influencing the product shelf life making it more valuable. It can be expressed in both wet and dry basis. The moisture content in wet basis, X_{wb} , is the ratio between the water content and the total mass in the material. The water content divided by the dried matter

contained in the sample is the moisture content in dry basis, X_{db} . The following equations represent the different relations for moisture content:

$$X_{wb} = \frac{m_t - m_d}{m_t} \quad (3)$$

$$X_{db} = \frac{m_t - m_d}{m_d} \quad (4)$$

$$X_{wb} = \frac{X_{db}}{1 + X_{db}} \quad (5)$$

where m_t is the total mass and m_d is the dried mass in each sample.

The initial and final moisture content for all tests carried out in the heat pump dryer were measured using the moisture analyzer “Mettler Toledo HB43-S”. After taking out the chamber its mass was measured every thirty minutes and moisture contents were calculated for the interval using the previous formulas (3, 4 and 5). The scale utilized to measure the mass change of the drying chamber containing the samples during the experiment was the “Mettler Toledo XP 600 2M DeltaRange”.

These relations were also used to determine the water content of the samples dried in the oven. In this case the final water content was given as an average of the three values obtained for each run.

4.4.2 Bulk and particle density

The bulk density ρ_b is defined as the average density of a volume of particles and voids in a specific medium, for instance air. It depends on factors such as the voids and compaction. On the other hand, the particle density ρ_p is the mass of one or several particles divided by the actual volume excluding voids.

The initial and final bulk densities were estimated dividing the initial and final mass by the respective volumes occupied by particles and voids in the cylindrical chamber.

Ten particles were selected from each material before the drying process and also after each test. The volume of each particle was calculated by measuring its width, length and height. The average mass and volume were used to obtain the initial and final particle densities.

4.4.3 Color

The color was measured before and after each drying process was finished. A “color difference meter XR948” was used. Prior to the measurement each sample was compressed and homogenized in a mortar to produce a representative sample to expose to the color meter. The color is given by three parameters that are brightness (L), red-green (a) and yellow-blue (b). The value ‘L’ indicates the amount of black (L=0) and white (L=100) while ‘a’ and ‘b’ measures the pure red and yellow ($a \rightarrow +a$, $b \rightarrow +b$) or pure green and blue ($a \rightarrow -a$, $b \rightarrow -b$).

4.4.4 Rehydration

Rehydration is a process that is often used as an indicator of the dried product quality. This method allows estimating the damage experienced by the raw material and product during the drying process. In rehydration the following processes occur: wetting of the surface, penetration of the water into the pores, adsorption on the internal surface of the matrix, diffusion into the solid matrix and equilibrium. Depending on the structure specific components may leach out of the product [1]. Even though different kinds of solvents can be used for the rehydration, the most common is water that is more bio-compatible than other substances that would transport important solids from the product including proteins, carbohydrates, lipids, minerals, etc.

The components of the system used for rehydration measurements are shown in Figure 15. For each test a disk of paper filter was weighed after being saturated with moisture and subjected to a slight vacuum. To perform these experiments, one and three units of the largest and smallest cubes respectively and one boiled particle were taken for each run. The dried samples were placed in a metallic net and immersed in a water bath that had been heated previously to 38°C. Every 5 minutes of rehydration, the samples were taken out of the water, wiped or drained and weighed to calculate the water mass gained by the product. Measurements were taken during 20 minutes.



Figure 15. Rehydration equipment

The porosity and shrinkage are highly related to the rehydration capacity. The porosity is defined as the empty volume contained in a sample, while the shrinkage refers to the volume change in one particle. They were calculated for the samples using the following formulas:

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad (6)$$

$$S_b = \frac{V}{V_0} \quad (7)$$

ε : porosity

ρ_b : bulk density (Kg/m³)

ρ_p : particle density (Kg/m³)

S_b : shrinkage

V : final volume (m³)

V_0 : initial volume (m³)

Chapter 5

5. Discussion and analysis

5.1 Moisture content and drying kinetics

5.1.1 Heat pump drying

The drying kinetics were determined for the eight tests carried out in the laboratory. The Figure 16 shows the resulting kinetic curves representing the moisture content in wet basis versus time.

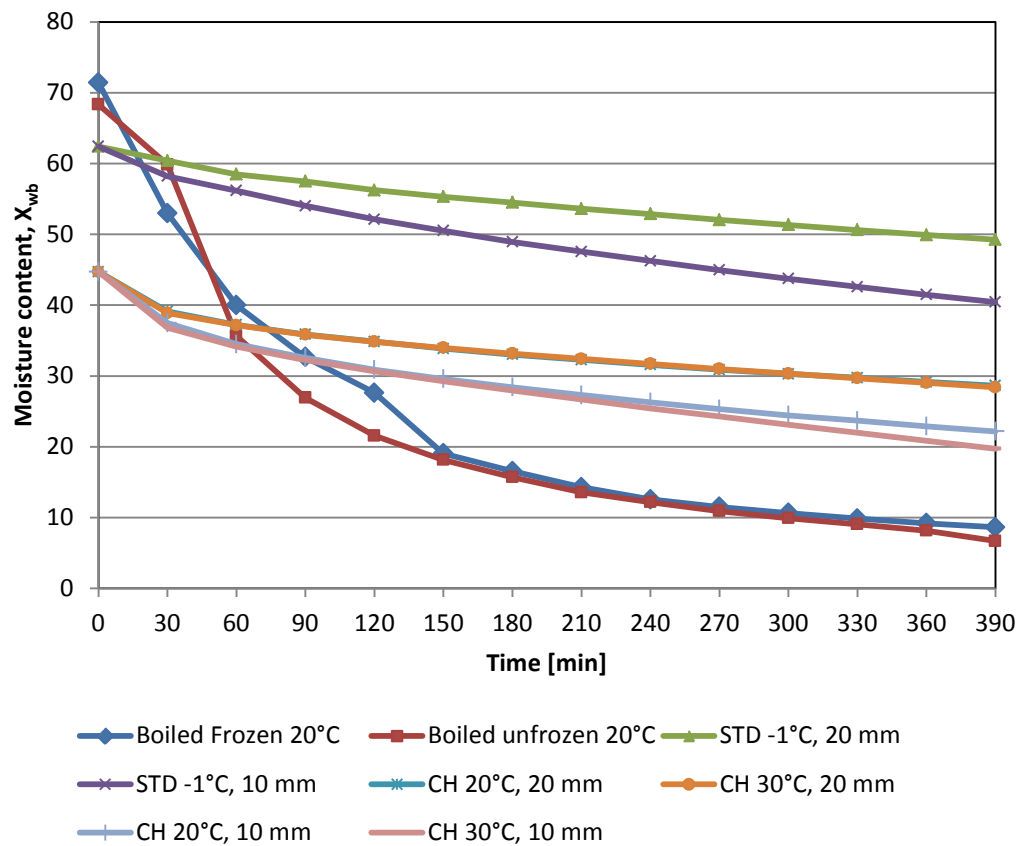


Figure 16. Moisture content kinetics for all tests

It can be seen that the curves tend asymptotically to the equilibrium moisture content. The highest initial moisture content is for the boiled samples with 70%wb, followed by the STD with 60%wb and the CH with 45%wb. Moreover, it can be observed that the final water content difference for the boiled material is about 60%wb while it is approximately 18%wb for the other tests. The moisture content versus time will be used to study the effect of different variables within the same material. A graphical procedure that gives a clear comparison of the drying rates for all tests is the plot of the drying ratio as function of drying time. This plot is shown in figure 17 by considering that near the end of the tests the equilibrium moisture content approaches 4%wb.

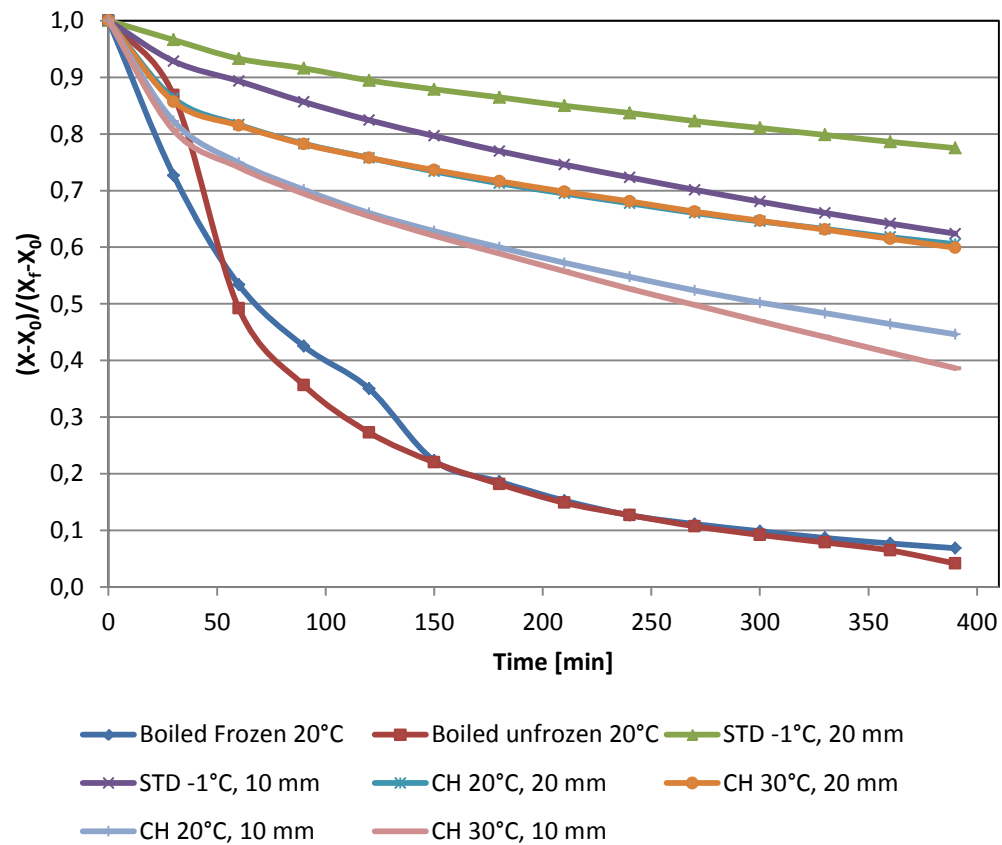


Figure 17. Drying kinetics for each test

The plot shows that higher drying rates are achieved for the boiled tests followed by the carbohydrate and standard samples. For the boiled material the slopes of the curves increase sharply until they reach a constant value after 180 minutes of drying. Initially, the ratio for the boiled frozen is slightly lower than for the unfrozen but after about 50 minutes it is reversed perhaps due to heating. The drying rates for the standard

samples are almost constant during the tests but higher for the smaller 10 mm and 20 mm samples. The carbohydrate tests are higher for the first 60 minutes. The succeeding plots present the parameter under study for each material separately to analyze the influence of shape, size and temperatures in the drying process.

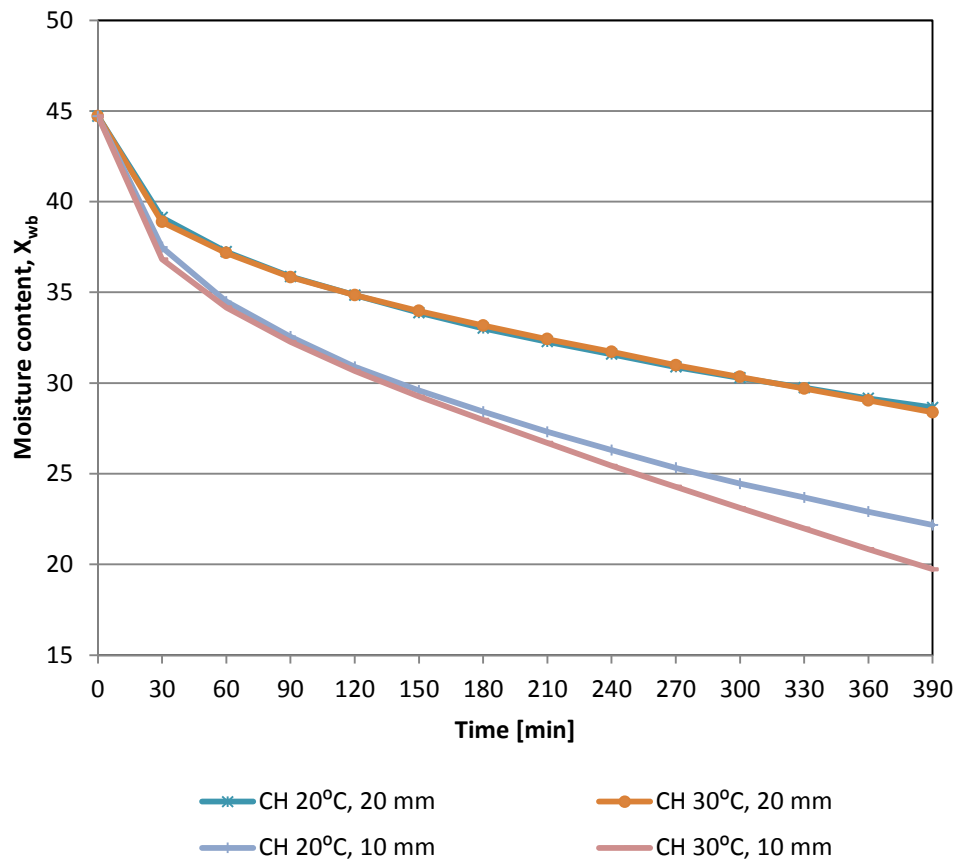


Figure 18. Moisture content of carbohydrate samples at different temperatures and shapes

Figure 18 depicts the performance of carbohydrate material tested under different temperatures and shapes. For temperatures of both 20°C and 30°C the drying kinetics curves obtained for the 20 mm cubes overlap. For the 10 mm cubes curves are similar but higher at 30°C. The curves for the 10 mm cubes at 30°C are higher than the curves for the 10 mm cubes at 20°C, indicating higher moisture content at the same time points.

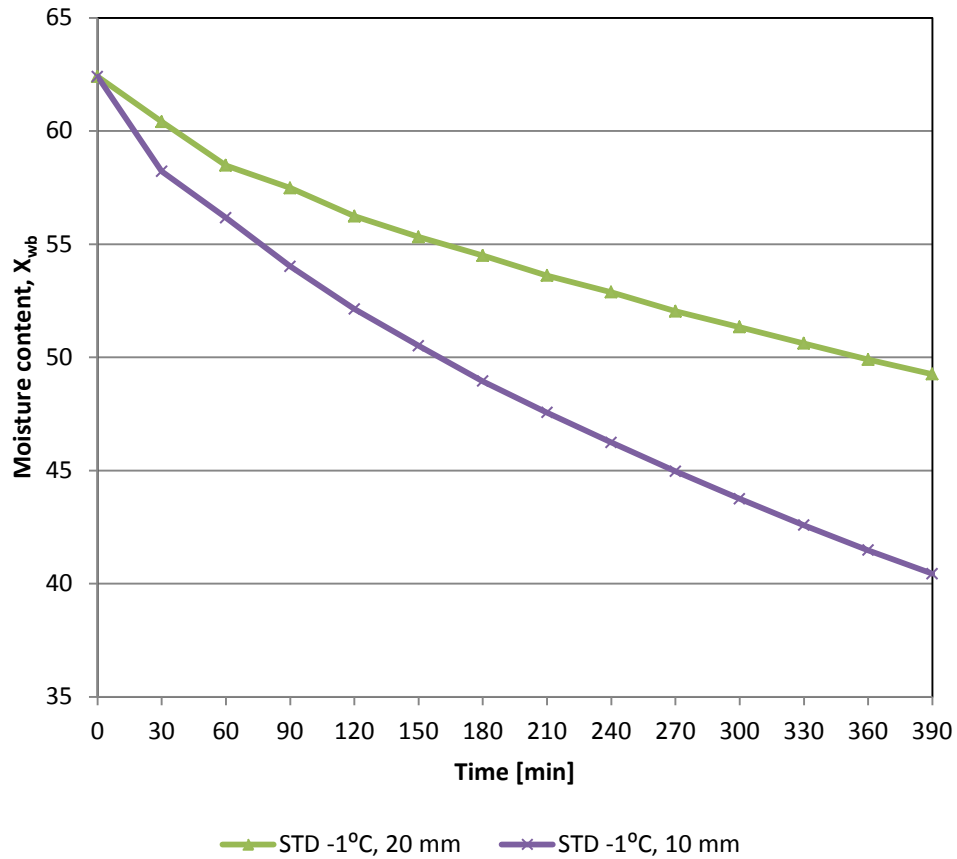


Figure 19. Moisture content of standard samples for different shapes

The Figure 19 shows that the moisture content decreases sharper for the standard material of 10 mm than for 20 mm cubes. At the end of the experiment the difference reached between both tests were about 10%wb.

These results confirm that mass and heat transfer enhance for the product that has been subjected to preheated treatments, the air temperature increases or size reduces. Heat transfer improves as the air temperature increases because the energy supplied in convection intensifies with the rise in the temperature difference between the surface and the drying air. A reduction in size has the similar effect due to drop in mass transfer resistance and the increment of the surface area for the smallest cubes.

The data and results also point out that the addition of carbohydrate affects and increases the drying rates. It also reduces the amount of water in the initial material since the addition of a dryer corn-starch fraction lowers water content in the mixture of starch and protein.

5.1.2 Oven method

Table 2 shows the moisture contents determined for each heat pump drying test using the oven method and comparison with the data obtained using the moisture analyzer. Except for numbers 6 and 7, the tests presented water content calculated by the oven method higher than the values from the moisture analyzer. Moreover, the differences between both values are larger for the standard particles of 10 mm size (6.89%) followed by the carbohydrate at 30°C and 10 mm (2.30%). The values for the boiled samples differ only by 0.28% on average. The reason for this is that the forced convection occurring in the drying chamber promotes better heat and mass transfer than the natural convection taking place in the oven.

N°	Empty mass (g)	+Wet	+Dried	m_w	$X_{wb} \%$	$\overline{X}_{wb} \%$	
						Oven	Analyzer
1a	2.583	8.169	7.669	0.500	8.951		
1b	2.580	8.172	7.665	0.507	9.067	8.997	8.640
1c	2.569	8.140	7.640	0.500	8.975		
2a	2.571	8.105	7.726	0.379	6.849		
2b	2.591	8.051	7.682	0.369	6.758	6.917	6.690
2c	2.585	8.109	7.714	0.395	7.151		
3a	2.561	8.130	5.280	2.850	51.176		
3b	2.601	7.963	5.251	2.712	50.578	51.687	49.010
3c	2.595	8.138	5.181	2.957	53.347		
4a	2.566	7.951	5.626	2.325	43.175		
4b	2.586	8.190	6.010	2.180	38.901	41.506	34.620
4c	2.593	7.911	5.647	2.264	42.572		
5a	2.586	8.875	6.895	1.980	31.484		
5b	2.578	8.828	6.978	1.850	29.600	30.768	29.310
5c	2.568	8.996	6.987	2.009	31.254		
6a	2.570	8.902	6.998	1.904	30.069		
6b	2.590	8.846	6.937	1.909	30.515	30.871	31.370
6c	2.585	9.078	6.996	2.082	32.065		
7a	2.586	9.058	7.511	1.547	23.903		
7b	2.578	8.976	7.390	1.586	24.789	23.585	26.170
7c	2.592	8.906	7.508	1.398	22.141		
8a	2.588	8.871	7.512	1.359	21.630		

8b	2.581	8.937	7.682	1.255	19.745	21.202	18.900
8c	2.570	8.951	7.527	1.424	22.316		

Table 2. Comparison between the oven method and the HPD

5.2 Bulk and particle density

The Figure 20 portrays the particle density before and after each drying experiment in blue and red bars respectively.

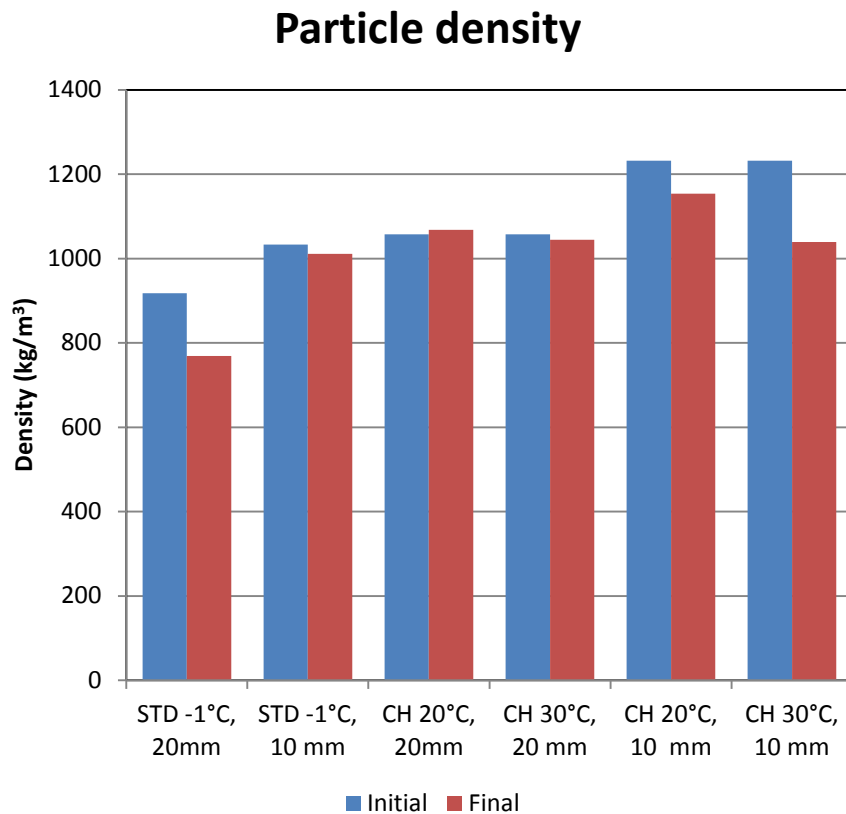


Figure 20. Particle densities

For the STD samples the initial and final particle densities may be expected to be the same since they originated from the same kind of material and are dried at the same temperature. However, the particle density is higher for the 10 mm cubes. This is because these dried samples are drier in the center and they become more compact. The initial particle density is also expected to be the same for all the experiments involving carbohydrates. As it happened for the STD samples, due to the internal moisture and compaction the particle density are higher for the smallest size. As the temperature rises the parameter under study gets lower and the result is more pronounced for the smallest pieces. For the same temperature the density varies more

and is higher for the 10 mm cubes. Besides this, the density for the CH samples is slightly higher than the STD samples. This is due to the internal moisture and higher compaction caused by addition of corn starch.

Figure 21 shows the initial and final bulk density for some of the drying tests. The highest values are reached for the carbohydrate samples followed by the standard and the boiled samples.

The initial bulk density for the carbohydrate samples is lower for the 20 mm than for the 10 mm cubic samples. This is because of the existence of more air gaps between the particles for the first case resulting in an increase of the volume for the same mass of product. For the 20 mm cubes the final density is higher than the density before drying. It happens because while the particles are being dried their sizes decrease and they fit and accommodate better into the measuring cylinder. Then the air gaps reduce and the volume drops in comparison with the mass loss. As the temperature increases, this phenomenon becomes more prominent and the density gets higher. For the 10 mm cubes the mass decrease is larger than the change in volume. As a result of it the final density drops. As the temperature grows the mass loss is higher and the bulk density decreases.

The boiled product follows the same behavior than the 20 mm carbohydrate samples while the standard sample has the same trend than the 10 mm cubes.

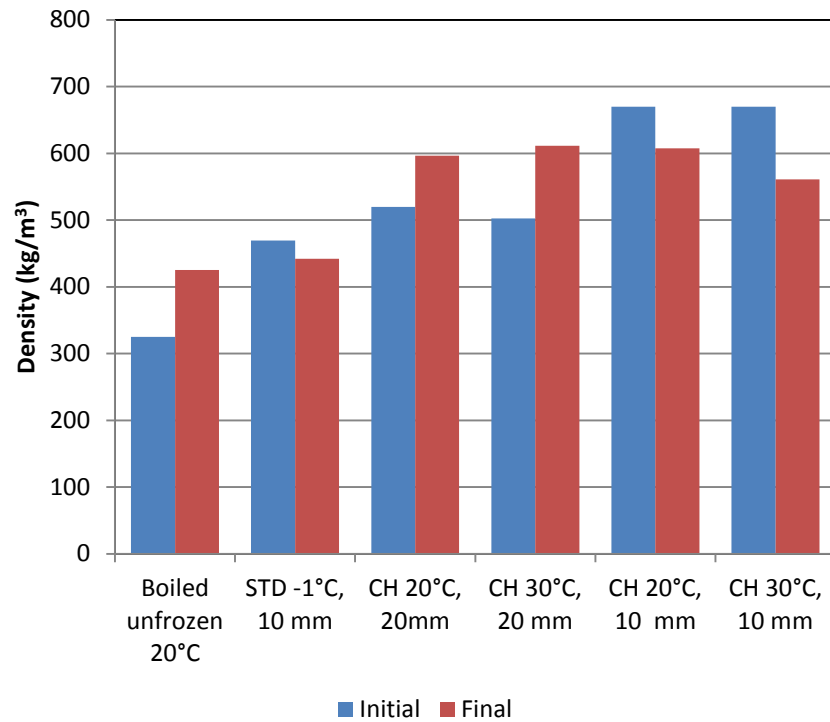


Figure 21. Bulk densities

The results on bulk and particle densities are summarized in Table 3. It shows numerically the percentage difference between the initial and final particle and bulk densities. The negative values indicate that it has been an increment in the final density in relation to the initial density.

	Particle density	Bulk density
Boiled frozen 20°C	-	-
Boiled unfrozen 20°C	-	-30.78
STD -1°C, 20mm	16.15	-
STD -1°C, 10 mm	2.11	5.80
CH 20°C, 20mm	-1.03	-14.75
CH 30°C, 20 mm	1.16	-21.71
CH 20°C, 10 mm	6.32	9.32
CH 30°C, 10 mm	15.63	16.23

Table 3. Particle and bulk density differences

The results and data confirm that heat and mass transfer improve for higher temperatures, smaller sizes and the inclusion of carbohydrate. On the other hand, the addition of carbohydrate leads to a higher density and it may influence transport costs.

5.3 Color

The different color parameters before and after each drying experiment with exception of the boiled samples were measured and the results are plotted in Figures 22 and 23. The initial color values are constant when the samples originate from the same type of material. The brightness measured with the L parameter decreases for every test. The initial L value is highest for the samples containing carbohydrates, which is a major contributor of the L color component. The next high value of L is for the standard and the boiled samples. The dried samples with carbohydrates had two zones distinguishable on their surfaces, a white and a red zone. As expected, the L parameter is larger in the white zone than in the red one. This is due to the concentration and migration of solutes on the surface caused by the evaporation of water and also because of some reactions that may occur in the product. The red color has not a trend although it increases in almost every experiment except for the experiments 3 and 5 in the white zone. The reason for which the red color turns into dark red increasing the 'a' value might be related to the destruction of the myoglobin through heat treatment but this needs verification in further research.

The yellow color measured as 'b' remains nearly the same for all heat pump dried samples.

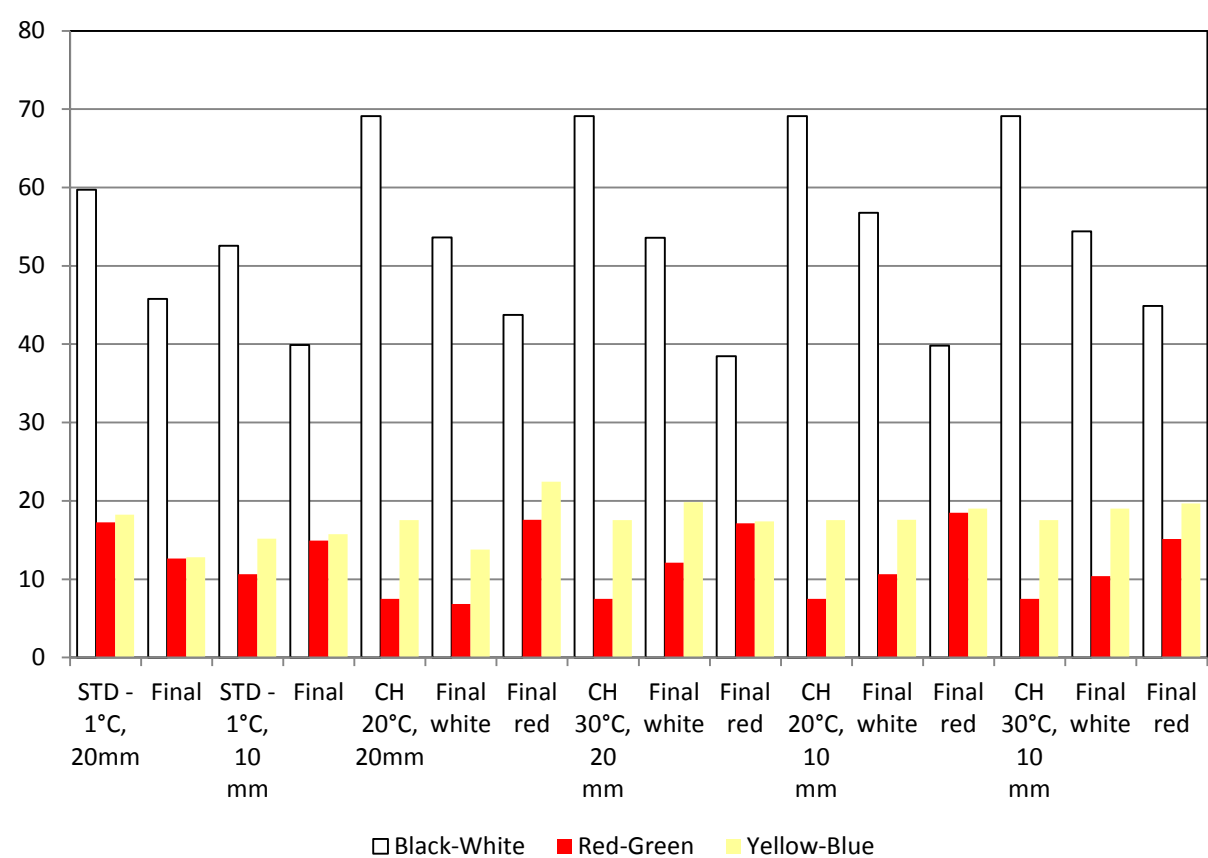


Figure 22. Color parameters

The plot in Figure 23 shows the evolution of the three color parameters for the carbohydrate samples separately to facilitate better understanding of the color differences between the white and red zones.

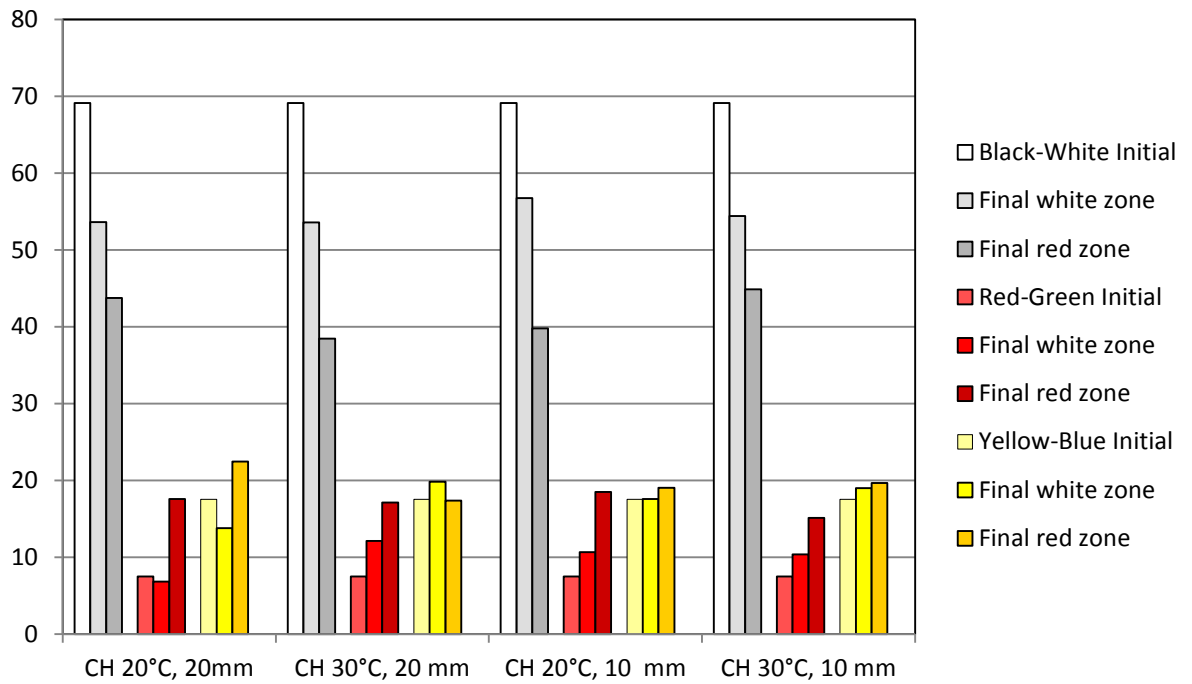


Figure 23. Color parameters for carbohydrate samples

The pictures for the tests 1 through 8 are shown in Figure 24 through 32. Figure 31 illustrates and shows that the dried carbohydrate pieces presented a lighter internal color.



Figure 24. Test 1



Figure 25. Test 2



Figure 26. Test 3



Figure 27. Test4



Figure 28. Test 5



Figure 29. Test 6



Figure 30. Test 7



Figure 31. Carbohydrate particles



Figure 32. Test 8

5.4 Rehydration

The rehydration measurements for all tests were done and the results are shown in Figures 33 through 36. The plots indicate that the water is initially quickly absorbed by the dried products. After a certain time, the water rate and intake trend tend to the equilibrium where the rehydration remains constant. The other plots show that a wide range of rehydration parameters can be reached. The lowest values are achieved for the carbohydrate samples in contrast with the highest values attained for the boiled products.

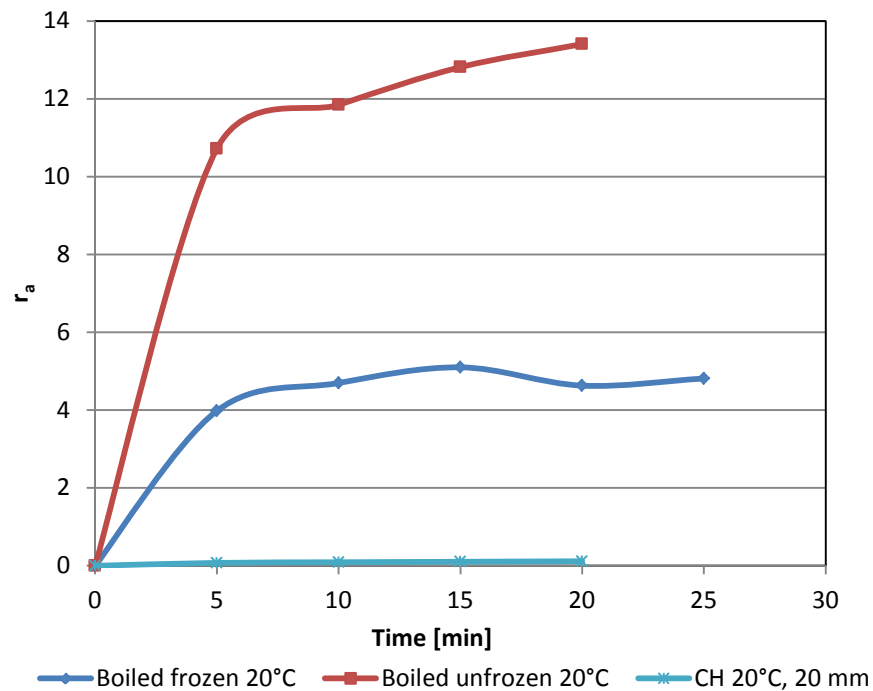


Figure 33. Rehydration capability

The rehydration for carbohydrate samples seems to increase as the drying temperature increases and as the product size drops. The cause is that the products dried at higher temperatures for the same size have lower shrinkage than samples dried at lower temperature. The 10 mm cubes have a higher rehydration rate due to its higher porosity and lower shrinkage. For the experiments dried at 30°C the mass reaches a constant value sooner than for those carried out at 20°C. Table 5.3 summarizes the results of the calculations of porosity and shrinkage for the carbohydrate samples.

	Particle density ρ_p (kg/m^3)		Bulk density ρ_b (kg/m^3)		Porosity $\varepsilon = 1 - \rho_b/\rho_p$		Shrinkage $S_b = V/V_0$
	Initial	Final	Initial	Final	Initial	Final	
CH 20°C, 20mm	1057.03	1067.91	519.72	596.40	0.508	0.442	0.786
CH 30°C, 20 mm	1057.03	1044.80	502.34	611.40	0.525	0.415	0.800
CH 20°C, 10 mm	1231.94	1154.10	669.80	607.40	0.456	0.474	0.829
CH 30°C, 10 mm	1231.94	1039.33	669.79	561.10	0.456	0.460	1.036

Table 4. Particle density, bulk density, porosity and shrinkage for the carbohydrate samples

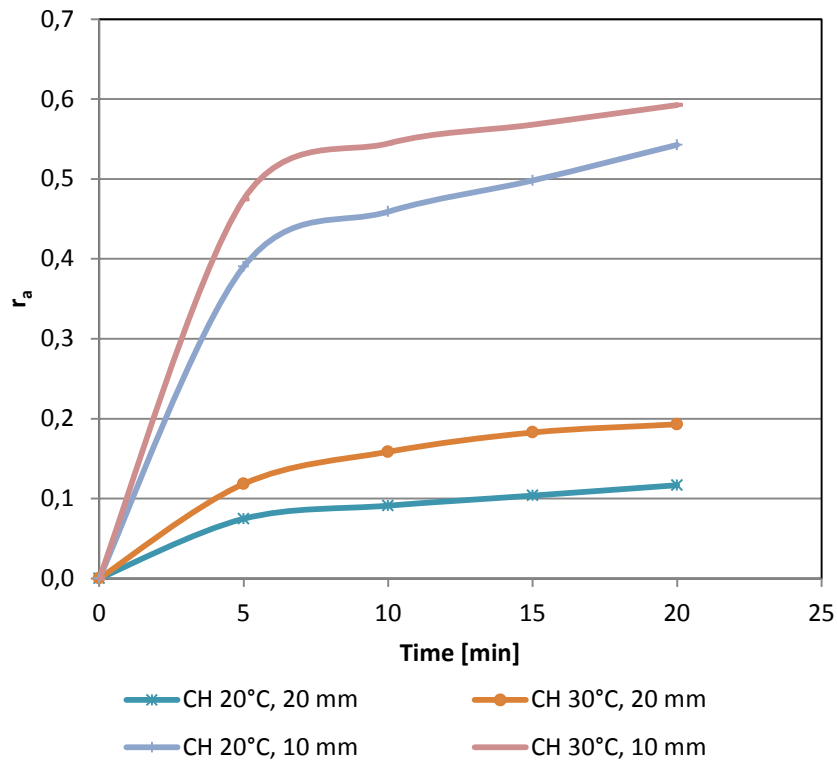


Figure 34. Rehydration of carbohydrates

The plot in Figure 35 allows comparison of the rehydration rate of STD samples at the same temperature for different sizes. The larger cubes sample reaches a constant value sooner than the smaller cubes. This is due to the large available surface area for absorption and lower shrinkage in the small cubes and for this kind of material. However, the difference between both ratios is relatively small.

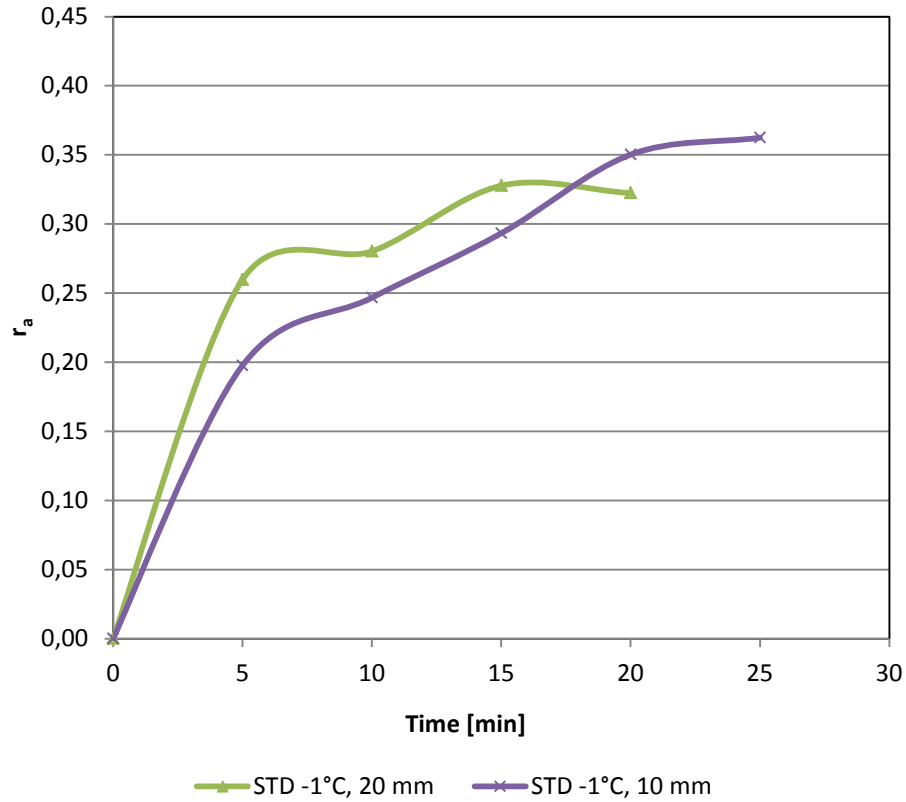


Figure 35. Rehydration of STD samples

The rehydration values for boiled samples fluctuate as Figure 36 shows. For the unfrozen sample in the first 5 minutes, the slope of the curve increases faster than for the frozen sample. After this, the rehydration for both samples remains almost constant and higher for the initially unfrozen sample. The rehydration ratio achieved for the initially unfrozen material is twice the value for the frozen sample.

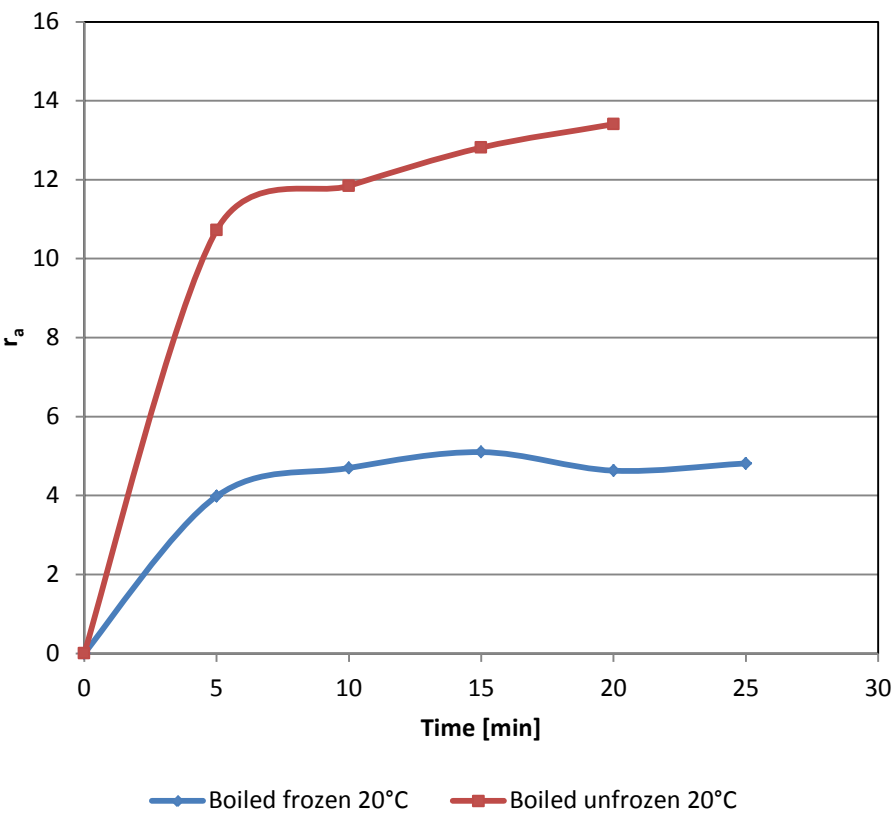


Figure 36. Rehydration of boiled samples

Chapter 6

6. Conclusion

6.1 Conclusion of the master thesis

Based on the experimental results it can be stated that the application of heat pump drying not only improve the drying rates but also enhances the quality of the final product. This has benefits in the production costs as well as increasing its value in the market due to a better aspect and appeal to the consumer.

The drying rate was influenced by the kind of material, the air temperature and the size or shape of the product. The highest values were for the boiled material without any differences for the materials that were initially frozen or unfrozen. Standard runs achieved lower drying rates than the carbohydrate samples, which thus had this benefit. The 20 mm pieces had reduced rates compared to the 10 mm pieces for both the standard and carbohydrate trials. The effect of the air temperature on the carbohydrate samples is that an increase of temperature leads to higher rates. However, the differences were very small.

The oven method results for initial moisture content of the raw materials were used to verify possible range by comparison with the calculated values based on mass change during drying. The moisture contents by oven method were higher with the largest differences for the 10 mm standard pieces (6.89%) and 10 mm carbohydrate at 30°C (2.3%) and lowest for the boiled unfrozen (0.23%).

The final particle and bulk densities were lower for higher temperatures and smaller sizes. The initial values of both densities were higher for the carbohydrate samples due to its larger compaction. The largest changes in the particle density were for the 20 mm

standard pieces (16.15%) and the 10 mm carbohydrate samples dried at 30°C (15.63%). The change for 20 mm carbohydrate particles was only a 1%. The bulk density differs more (30.78%) for the boiled and unfrozen sample and less (5.80%) for the 10 mm standard run.

The analysis of the color parameters indicates that after drying the 'L' value decreases for all tests. The 'a' value increased in nearly every run and the 'b' value remained constant. The carbohydrate samples presented two zones, one was bright and another was dark red. The internal parts of these particles were almost white.

The rehydration rate of carbohydrate enhances with the increment of drying temperature and with the reduction in size. For the standard material, the rehydration parameter was higher for the larger cubes although the difference was small. For the boiled and initially unfrozen samples the rehydration achieved was twice higher than for the frozen sample. The carbohydrate sample dried at 30°C had lower shrinkage values for materials with the same size. The smallest particles presented higher porosity and less shrinkage leading to higher drying rates for these samples.

6.2 Further research

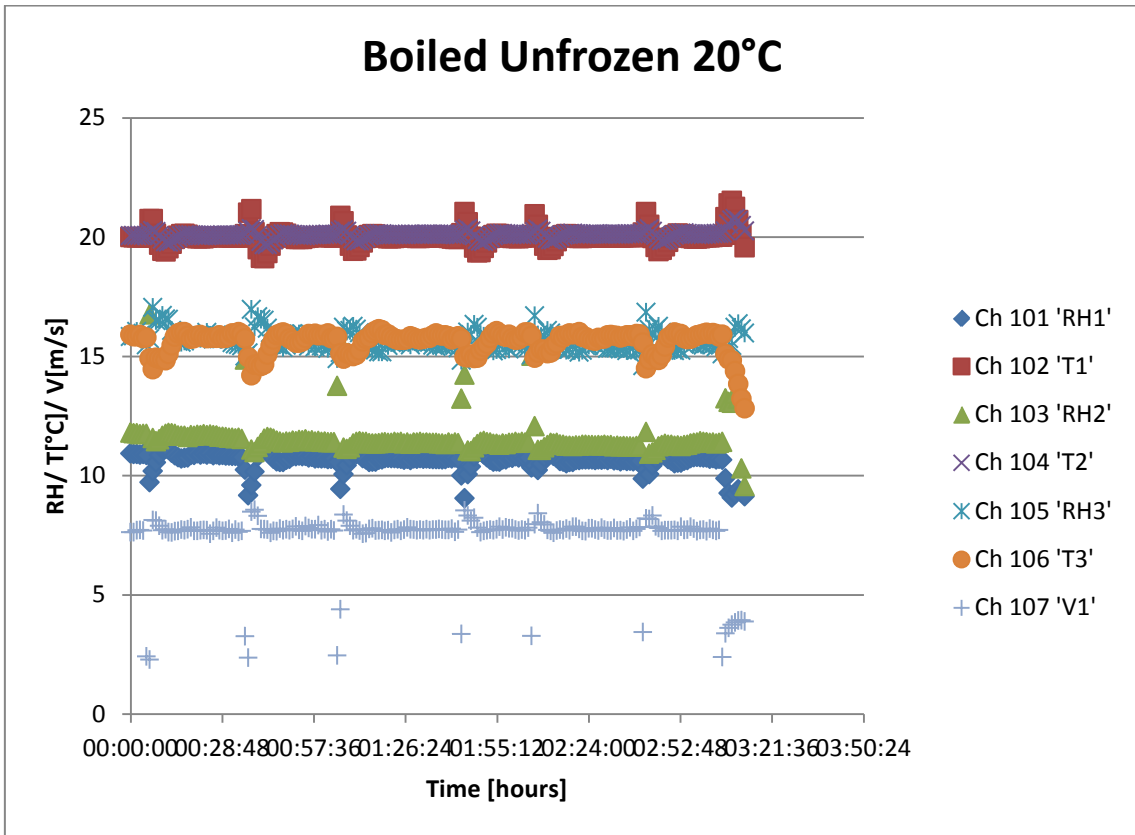
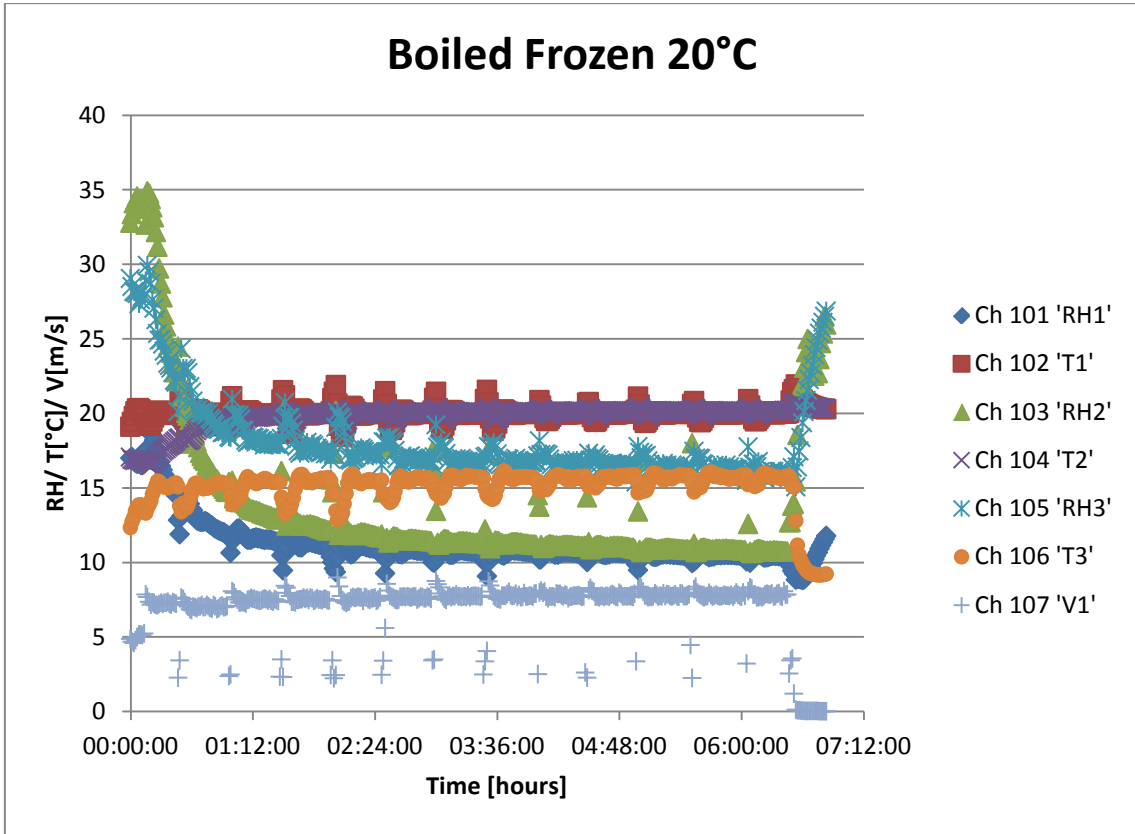
Quality properties of food are very important for the reasons that have been outlined before. More experiments are recommended to confirm the optimal drying conditions providing a final dried product with a better quality and properties.

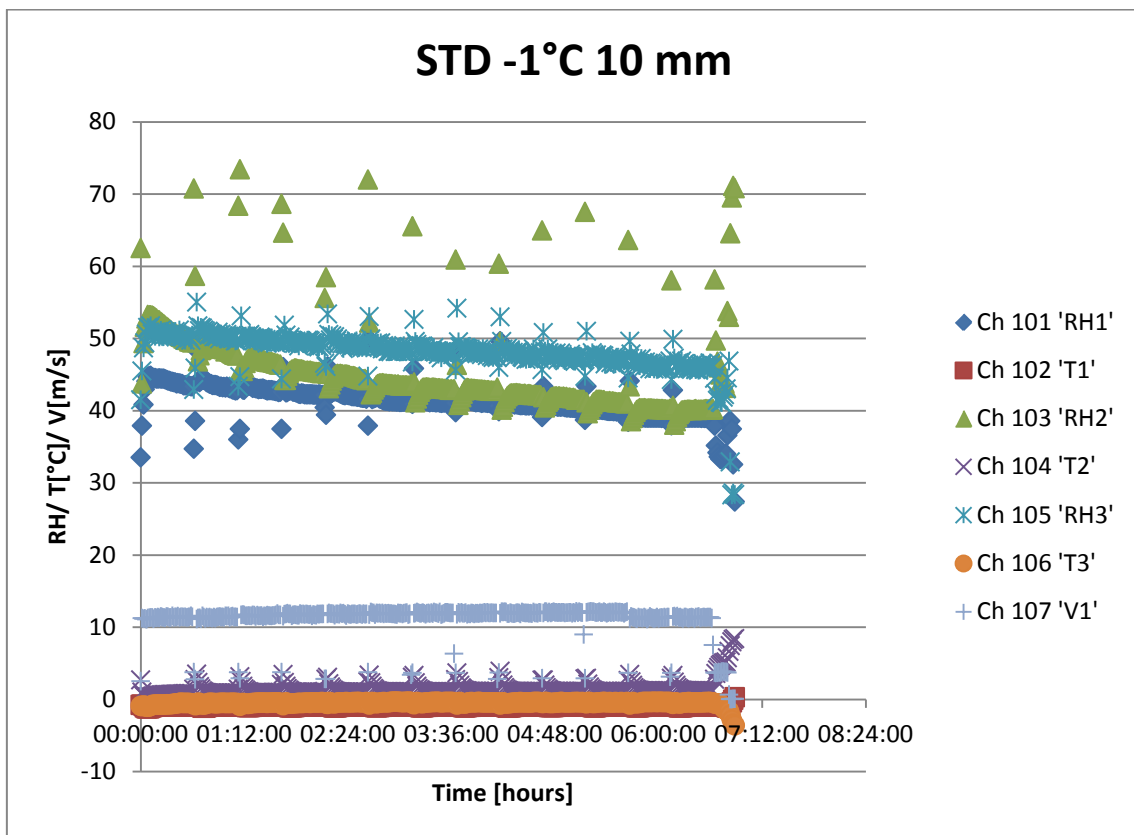
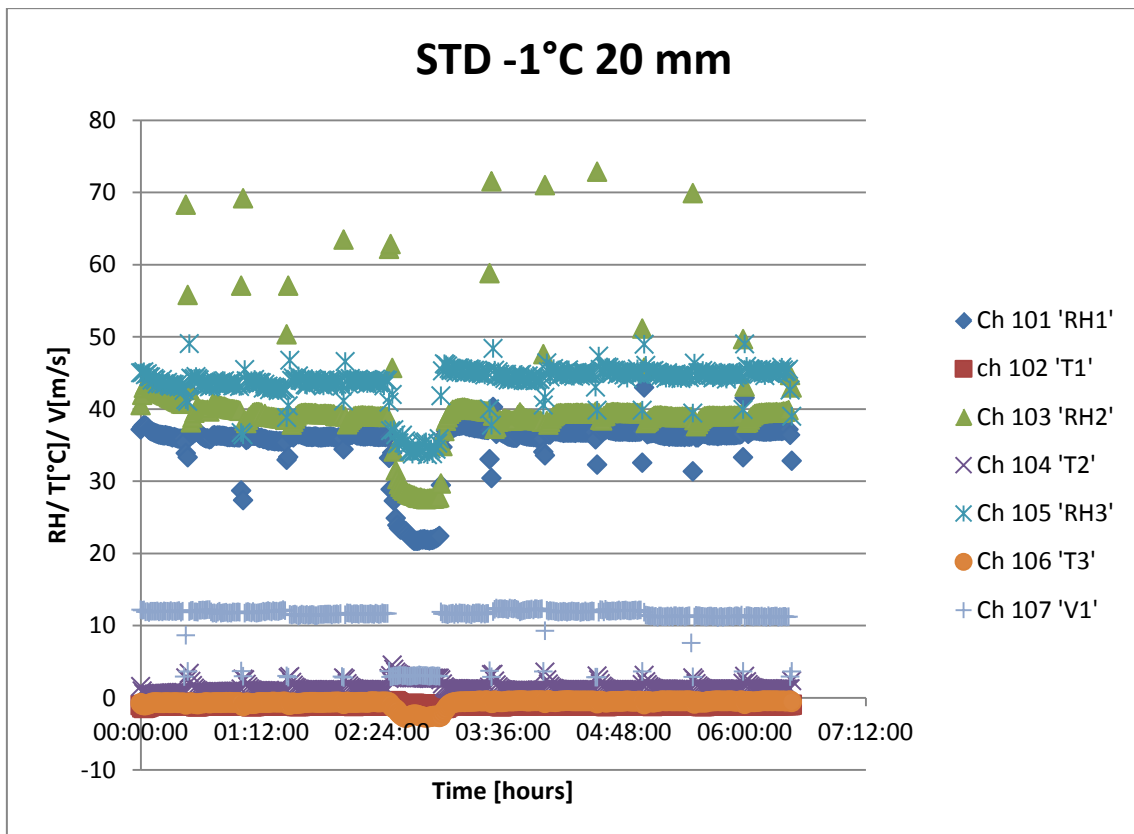
A wide range of temperatures could be applied to find better drying rates while keeping the quality characteristics. Different combinations of air velocities may improve the rates of heat and mass transfer between the product and the air flow in the drying chamber. Moreover, experiments to better understand why the red color turns into dark red and the reactions that may occur within the product could be done. Other shapes should be also tested, for instance, spheres. For this geometry both processes of heat and mass transfer would be enhanced due to its largest contact area.

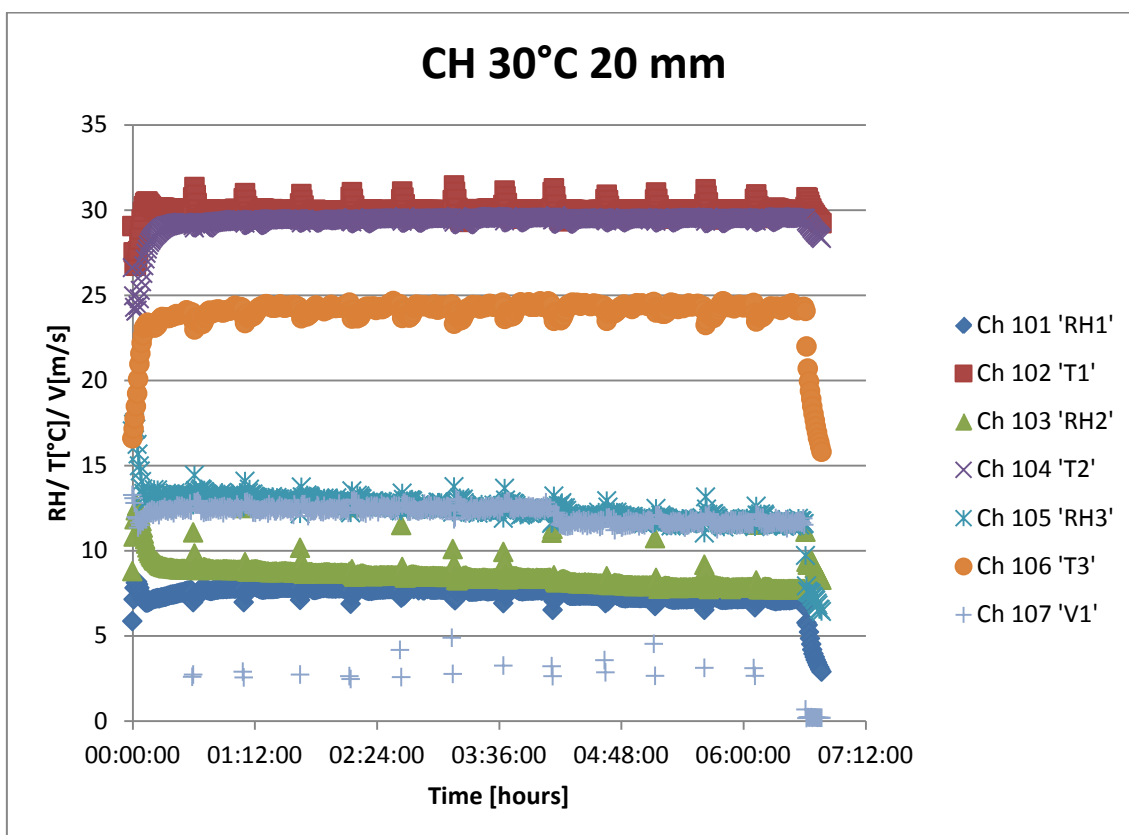
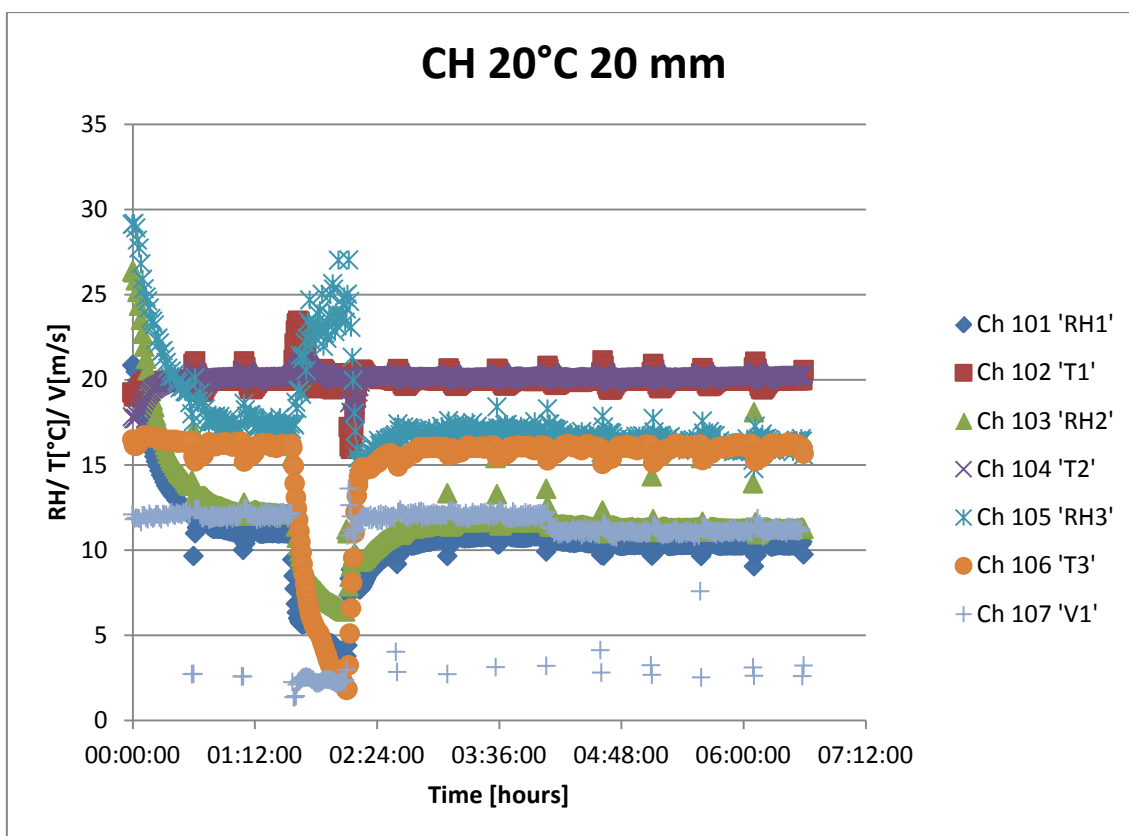
Carbohydrates are added to some animal feed as a substitute of proteins to reduce its price. They supply an important amount of energy but there is limitation on quantities and health effects. Taking this into account experiments involving different masses of carbohydrate could be carried out to determine which are the best fractions and proper conditions giving high quality product and minimum costs.

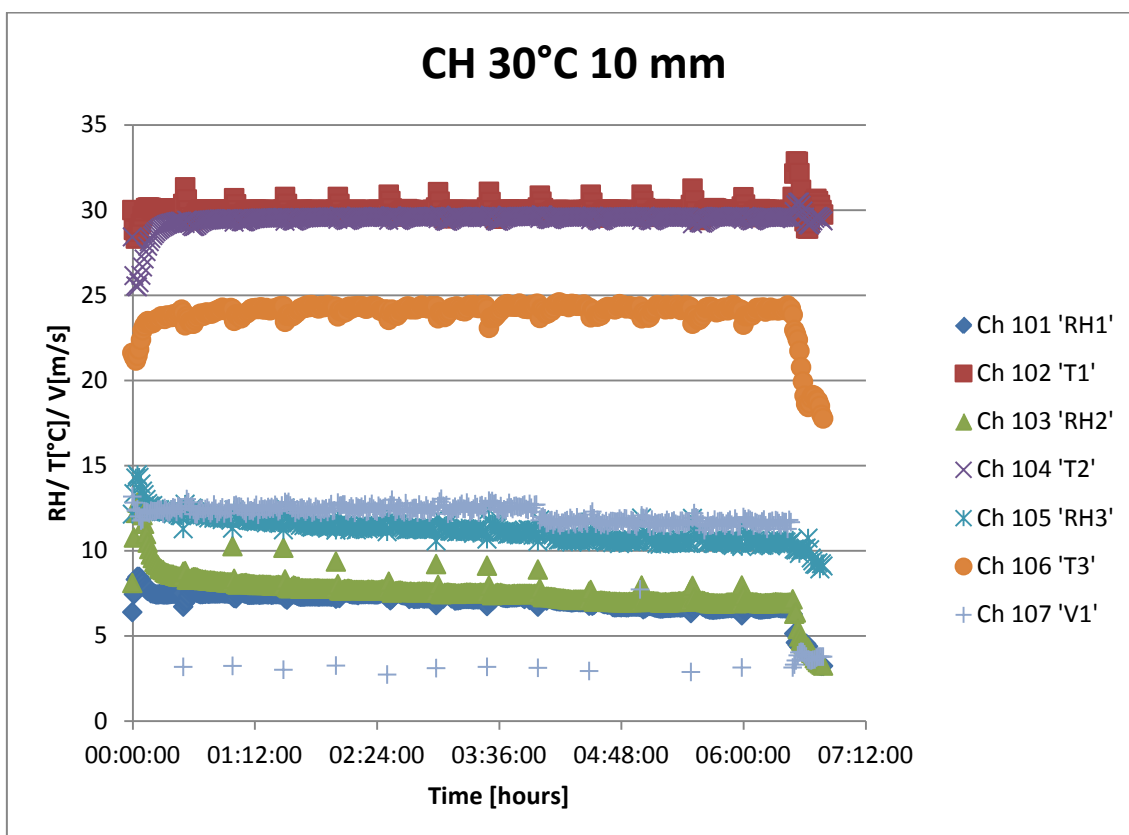
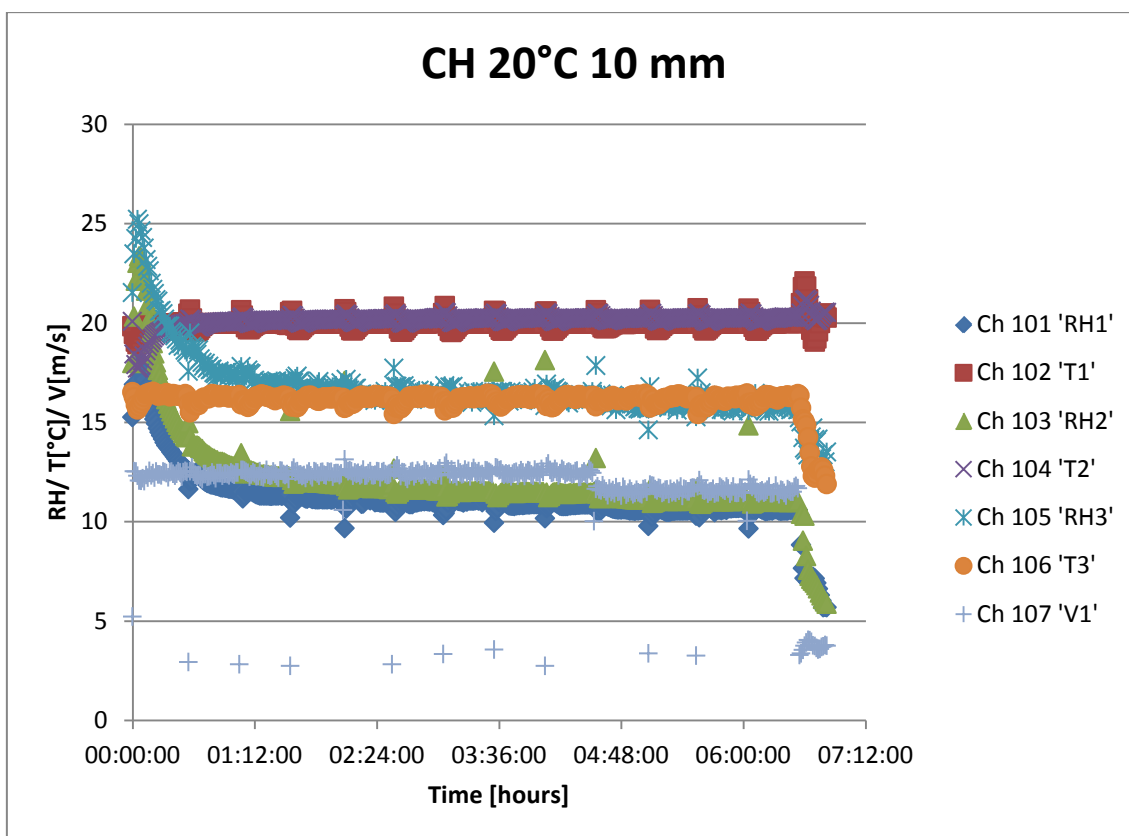
APPENDIX

Parameter values during the performance of the eight experiments in the laboratory









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